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#### PATENT SPECIFICATION

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(71) We, ROLLS-ROYCE (1971)
LIMITED, a British Company of 14-15 Conduit Street, London, WIA 4EY, formerly of 1 Bank Building, Princes Street, London EC2, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed to be particularly described in and by the following statement:—

This invention relates to the protection of surfaces in the vicinity of such operations as fusion welding, flame spraying or laser cutting where spatter is produced, from contamination

by such spatier.

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Fusion welding is a generic term covering forms of welding in which the weld is carried out solely by the melting of the metals to be joined. Electron beam welding is an example of such a form of welding.

It is found during such methods of welding, that some spattering from the molten metal often occurs. Such spattering metal readily adheres to surfaces in the vicinity of the welding operation and is normally very difficult to remove. Hitherto expensive and inconvenient operations such as grinding or machining have been utilised to restore the spatter contaminated surfaces to their original condition. It is an object of the present invention to provide a method of fusion welding in which surfaces in the vicinity of the welding operation are protected from the molten products thereof.

Flame spraying is the term used to describe a method of depositing finely divided metal and/or refractory particles in the heated, semi-molten condition to form an adherent coating on a suitable surface. The particles may be heated by means of a plasma, or by the combustion of such gases as oxygen and acetylene. It is often necessary when flame spraying to protect or mask surfaces in the immediate vicinity of the surface to be coated so as to prevent their contamination by

the particles being sprayed. It is a further object of the present invention to provide a method of flame apraying in which surfaces in the vicinity of the flame spraying operation are protected from the semi-molten products there-

Laser beam cutting is a method of cutting metals, ceramics etc. in which a laser beam is used to melt a small region of an article so as to produce a cutting effect. The spattering produced by such melting is liable to contaminate surfaces in the vicinity of the cut. It is yet a further object of the present invention to provide a method of laser beam cutting in which surfaces in the vicinity of the cutting operation are protected from the molten products thereof.

Thus, according to the present invention, a method of protecting at least part of at least one surface which is positioned or is intended to be placed in the vicinity of a metal and/or refractory spatter-producing process from contamination by the spattered molten or semi-molten products of such a process comprises the steps of costing said at least part of at least one surface with at least one layer of a composition comprising a mixture of an 70 organophilic cation-modified clay (as hereinafter defined) and a volatile organic vehicle, removing substantially all of said vehicle from said coating to leave a film of said clay on said at least one surface, effecting said spatterproducing process, and removing any of said sparter together with said film.

The term metal as used herein includes alloys.

The term "organophilic cation-modified clay" as used herein means a clay modified by covering the greater part of the surface of the particles of the clay with alkyl or aryl organic radicals coupled to the clay ionically by means of an onium base.

An onium compound has been defined in

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[Price 33p]

Hackh's Chemical Dictionary-Second Edition-as "A group of organic compounds of the type RXHy which are isologs of ammonium and which contain the element X in its highest valency; where X is pentavalent as in ammonium, phosphonium, arsonium and stibonium, where X is retravalent as in oxonium, sulphonium, selenonium and stanonium compounds and, where X is trivalent, as in iodonium compounds".

They may be considered as addition compounds, this being further described under the heading of oxonium, carbonium, stibonium,

inium, and — ylium.

The cation-modified clay may be produced for example by converting the organic bases the salts formed by the addiacid such 25 hydroof tion ឧព chloric acid or acetic acid dissolved in water, 20 and adding the selected onium compounds to

an aqueous clay dispersion.

The clays to be used in the preparation of the organophilic cation-modified clay have an exchangeable inorganic cation and are pre-25 ferably those which originally exhibit a base exchange capacity of at least 40 millilitres per 100 grams of the clay, for example the bentonite group of clays which are composed principally of minerals of the montmorillonite group (including monuforillonite, hectorite, saponite and montronite) are particularly suitable. Other base exchange materials may however be used.

Furthermore, an organophilic cation-modi-35 fied clay which may be used in accordance with the invention is one obtained by reacting a clay, in which at less some of the exchangeable ions, other than sodium, have been replaced by sodium ions, with an onium com-

40 pound.

Details of the preparation of organophilic cation-modified clays are given in United Kingdom Specifications Nos. 664,830, 782,724 and 904,880 and in U.S.A. Specification No.

2,531,440.

Examples of organophilic \*cation-modified clays which may be used are dimethyldioctadecyl ammonium montmorillonite, dodecylamine montmorillonite and dimethyldiocta-

50 decyl ammonium hectorite.

The volatile liquid organic factilitates the application and deposition of a film of the organophilic clay on the surface to be protected. As the vehicle is to be removed (from the coating composition applied to the surface to be protected) prior to the commencement of the spetter producing process, organic vehicles of low and medium boiling point are preferred to those of high boiling point. In general, liquid organic vehicles having boiling points of below 150° C at atmospheric pressure are preferred. Among liquid organic vehicles of low boiling point (i.e. boiling under 100° C at atmospheric pressure) mention may be made of aliphatic and alicyclic compounds (including halogenat-. ed compounds), such as ethyl alcohol (including methylated spirit), methyl acetate, butyl chloride, trichloroethylene, 1:1:i-trichloroethane, acetone and cyclohexane, as well as aromatic compounds such as benzene and petroleum naphtha. Medium boiling point organic vehicles (i.e. those boiling between 100 and 150° C at atmospheric pressure) including n-amyl a.cohol, n-butyl acetate and cyclobexanone as well as toluene, xylenes, monochlorobenzenes and tetrahydrofurfuryl alcohol. High boiling point organic vehicles including acetonyl acetonate, butyl butyrate and benzyl alcohol. Examples of other volatile liquid organic vehicles suitable for use in accordance with the invention are given in "Industrial Solvents" by I. Melan (1950) p. 36 to

It may be possible in practice to use two

or more volatile vehicles.

Preferably the coating composition also contains an additive which is a non-volatile organic substance having adhesive properties and capable of forming a dry film when mixed with the organophilic cation-modified clay.

As examples of said non-volatile organic

additives, mention may be made of:

synthetic resins, such as acrylic resins (e.g. "Bedacryl" 122X manufactured by Imperial Chemical Industries Ltd.), the word "Beda-cryl" being a Registered Trade Mark, alkyl resins, (e.g. "Epok" C.460 manufactured by British Resin Product Ltd.), the word "Epok" being a Registered Trade Mark, silicone resins (e.g. M.S. 805, manufactured by Midland 100 Silicones Ltd.), and chlorinated rubber;

natural resins, such as resin, dammar and

shellacs:

synthetic waxes, such as the Seekay waxes ("Seekay" is a registered Trade Mark) sold 105 by Imperial Chemical Industries Ltd., Abril waxes sold by Abril Industrial Waxes Ltd., and chlorinated polyethylene;

natural waxes, such as carnauba wax, shellac

wax, palm and corton seed waxes;

organic substances capable of forming a dry film, such as solid esturated and unsaturated carboxylic acid (e.g. lauric, stearic, m-toluic, sebacic and fumaric acids), solid esters of saturated and unsaturated carboxylic acids (e.g. 115 ditetradecyl sebacate) solid alcubols and glycerides (e.g. cetyl alcohol, \(\beta\)-monopalmitin and a-monostearin); mention may also be made of halogenated polynuclear compounds for example, bi- and polyphenuls (e.g. the mixtures sold by Monsanto Ltd. under the Registered Trade Mark "Aroclor");

liquid organic esters capable of forming a dry film and drying and semi-drying oils, e.g. cotton seed oil. More than one such binder may be used in the coating composition if desired and it may often be convenient to introduce the binder into the other components of the coating in an organic solvent, particularly when the commercially available form of the 130

binder comprises the binder in an organic solvent (as is the case, for example, with "Bed-

acryl" 122X).

The coating composition may also contain
an additive which is a non-volatile simple or
complex refractory oxide having filling properties and capable of forming a dry film when
mixed with the organophilic cation-modified
clay. The addition of such an oxide improves
the covering power of the composition. More
than one such refractory oxide may be added
to the coating composition if desired.

The proportions of said clay, volatile vehicle, non-volatile additive and non-volatile 15 retractory oxide additive to be employed in coating compositions to be used according to the method of the invention may be varied within wide limits, preferably 2 to 50 parts by weight of said organophilic cation-modified 20 clay, 50 to 300 parts by weight of volatile vehicle, 0 to 50 by weight of non-volatile additive and from 0 to 30 parts by weight of non-volatile refractory oxide additive. The criterion in determining these portions is the 25 ease with which an adequate film of the organophilic cation-modified clay (and the nen-volatile additive and non-volatile refracting oxide additive when present) is formed on the metal surface. Generally speaking the film of the dried coating should not be less than 5 microns in thickness, but preferably is from 15 to 40 microns. In practice however it is usually

more convenient to apply a minimum number of coatings to the metal and a coating composition having a concentration of about 200 grams of organophilic cation-modified clay per litre of volatile organic vehicle may be used with advantage.

In processes according to the invention in which a non-volatile additive forms part of the coating composition, good results have been obtained when the ratio of the weight of said organophilic cation-modified clay to the weight of said non-volatile organic additive is from 1:1 to 1:4.

The following method of preparation was used for the coating composition in the exam-

ples:
A weight of the organophilic cation-modified clay (constituent (a)) was milled with, a weight of oil soluble dye (if used) (constituent (b)) as a witness of coverage a weight of non-volatile refractory oxide additive (if ured) (constituent (e)) and, three fifths of the weight of the volatile liquid organic vehicle (constituent (c)) in a stundess steel ball mill for four hours. The mixture was diluted with the remaining liquid organic vehicle and a weight of non-volatile organic adhesive substance (if used) (constituent (d)). The mixture was further milled for half an hour.

The following table shows the constituents and proportions in the coating compositions used in the examples:

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. Con. - abbievrated for Constituent.

Organic stances (d)	W.C.vol	Se gras	SO cms	25 mis	,	50 gms	So gras	35 gras +49 gms	
Non Volatile Organic Adhesive Substances (d)	Type	Bendacryl 122X	Bendacryl 122X	M-: 805		"Aroclor" . 268	m-toluic acid	Stearic acid + Pedacryl 144TL	
)r <b>g</b> anic	Wt. vol	1000 gms	1000 gms	\$00 mls	1000 mls	1000m1s	1000 mls	1460 gms	100 gms
Volatile Liquid Organic Vehicle (1)	Type	Isopropyl Aicohal	Isopopyl Alcohol	Toluene	Toluene	Toluene	Tolucne	Trichioroethy lene	Isopropy! Alcohol
(a) 3	ž	ı	2 gas	<b>5.</b>	2 gms	3 E3 5	8 (1) 8	ng l	2 gms
O.1 Soluble Dye (b)	ják;	,	Waroline Red	Naxoline Red	Waxoline Red	Wascline Red	Vatoline Red	Waxuline Greed	Vaxoline Red
<u></u>	- 3	.300 gm.s	\$ \$ £ £	1.5 gras	8E 0.	30 gms	30 gm>	Start Oy.	250 gms
Organophics Modified Clay	1) pe	Dimethy (divertude cy.) Animonium Montmorilli- onite (Bentone 34)	Dimethy Idioctadecy   Ammonium Montmorill- onite (Bentone 34)	Dimethy Idioctadecyl Ammoniun, Montmorilli- onite (Bentone 34)	Zeolite Clax (Bentone 37)	Dimethyldisetadecyl Ammonium Montmorilli onite (Bentone 34)	Directhyldiss tadesyl Americanium Montrocilli- onice (Bentone 34)	Dimethyldroctadecyl Ammonium Montmorilli- onite (Bertone 34)	Dimethy Piroctade cyl Ammonium Montmotilli- onite (Bentone 34)
Con.	N.	_	· ·	<b></b> .	-	·	c	_	æ

Con.•	Organophilic Medified Clay	ly	Oi! Soluble Dye (h)	(A) 0/Q	Volatile Liquid Organic Vehicle (c)	Organic	Non Volatile Organic Adhesive Substances (d)	Organic lances (d)
Š.	Tipe	i,g	1 ype	į.	Type	Wt 'vol	Type	We'val
3	Dimethyldioctadecyl Ammonium Mintmotril- onite (Bentone 34)	100 gms	Manoline Red	2 gms	Trichioroethy lene	1000 gans	·	1

Con	Organophilic Medified Clay		Oil Soluble Die	کے	Volatile Liquid Organic Vehicle	I C Ic	Nen-Volatile Organic Adhesive Substance	rganic	Refractory Non-	Non-
Ň.	Type	=	Type	i a	7,78	*	Type	1,4	Type	13
01	Diemthy Idioctadeey ! Anniwitium Montmort Horite (Bentone 34)	<b>9</b> 051	Naxoline Red 2 gms	É	Buty I. Alcohol	1000 gas	Bedsery   122X	See Day	50 gms Titanium Dioxide	100 8ms
=	Bentine 34	3002		1	I so-ptopy i	1000		t	Zirconium	25
									31116	SE SO

Preparation (1) was prepared according to the previously described method. Erample 1.

Two recumpular blanks of the nickel alloy known as Nimonic 80 were treated as fed-1

greasy, were paraffin washed and then de-greased in trichloroethicus vapor to remove organic materials. Paint was removed by suit-able puint stripper. 1. The metal surfaces, being particularly 2

was masked with a marking tape suitable for dry grit blasting. The other half of the side of each blank was blasted with a dry 18/24 The masking was then removed and the re-2. An area of half of one side of each blank mesh aluminium oxide grit at 30 p.s.i.

\* 7 }

unblasted area on one of the specimens was 3. As soon as possible after drying, the

2 R maining adhesive removed by wiping with an actions south cloth, and allowed to dry. painted with one coat of the mixture prepared as above. The coating was allowed to dry in air for 10 minutes. The other specimen

was left uncoated.

4. One of the specimens was pre-heated by the flame of a plasma spray gun (3MB equipment, obtainable from Metco Ltd). The powder feed was switched on and tungsten carbide/cobalt composite powder (Stellundum 52F obtainable from Deloro Stellite Ltd) was sprayed on to the blasted area, but up to the edge so an even coating was obtained all over the blasted area. This was continued until an even coating of 0.006" was produced.

The whole of this operation was repeated

for the other specimen.

5. On examination after cooling the specimen coated with the mixture was observed to have some sprayed coating adhering to the 20 masked area. This was easily scraped off with the blade of a scalpel, and the specimen was examined again. None of the sprayed coating was seen to adhere to the masked area.

The unmasked specimen had sprayed coating adhering to the unblasted area. This could not be removed with the blade of a scroel.

Example 2. .

Preparation (2) was prepared according to the previously described method. In this example, two blanks of a martensitic steel known as "Jethete" ("Jethete" is a registered Trade Mark) were used and the preparation technique was substantially similar to that in

The spraying was done with an oxygen/ acetylene flame spray gun (Metco 10E) and a nickel/aluminium wire (Metco 405) was the

sprayed medium.

In all other respects, including the results of the examination, this example is similar to Example 1.

Example 3.

Preparation (3) was prepared according to the previously described method. The following

45 procedure was then adapted.

1. Referring to the drawing, which is by way of example only, two cylindrical hollow test pieces 10, 12 of electron beam weldable steel were degreased by being paraffin washed 50 and then exposed to trichloroethylene vapour.

2. As soon as possible after step 1, the inner surfaces of the test pieces 10. 12 were sprayed with one coat of the couting mixture in the region indicated at 14. The illustrated thick-55 ness of the coating is of course greatly exaggerated. The coating was allowed to dry in air for 10 minutes.

3. The test pieces were butt-welded together by means of conventional electron beam weld-60 ing apparatus 16, which was rotated about the longitudinal axis 18 of the test pieces 10, 12. A weld bead 20 was formed on the inner surface of the weld, indicating that the welding operation was proceeding satisfactorily and being effected over the full thickness of the walls of the test pieces. Weld spatter was produced and was observed to be projected from the site of the welding operation across the inside of the test pieces to the opposite surfaces thereof as indicated by the arrows 22.

4. After the welding operation was completed, the last pieces were parmitted to cool, and it was found that the weld spatter which had been projected on to the coated region 14 of the surfaces of the test pieces could be removed, together with the coating, by wiping

with a cloth.

Example 4.

Preparation (4) was prepared according to the previously described method. Two hollow cylindrical test pieces of an electron beam weldable titanium/copper alloy containing 2% of copper and the halance titanium, except for trace impurities, were electron beam welded together as described in Example 3, except that the mixture of the present example was used instead of the coating mixture of Example 3. The test pieces were degreesed by means of the alkaline degreeser "Orthosil" made by the Stavely Iron and Chemical Co. Ltd. It was found that the weld spatter could be removed together with the coating 14 by wiping with a cloth.

Example 5.

Preparation (5) was prepared according to the previously described method. Two hollow cylindrical test pieces of electron beam weldable stainless steel containing 18%, chromium and 8% nickel were electron beam welded together as described in Example 1, except that 100 the coating mixture of the present example was used instead of the coating mixture of Example 1. It was found that the weld spatter could be removed by wiping with a cloth.

Example 6. Preparation (6) was prepared according to the previously described method. Two hollow cylindrical test pieces of electron beam weldable Nimonic 73 alloy ("Nimonic" is a registered Trade Mark) were electron heart welded 110 together as described in Example 1, except that the coating mixture of the present example was used instead of the coating mixture of Example 1. It was found that the weld spatter could be removed by wiping with a cloth.

Example 7.

Preparation (7) was prepared according to the previously described method. Two hollow cylindrical test pieces of electron beam weldable stainless steel containing 18% chromium 120 and 8% nicked were electron beam welded together as described in Example 1 except that the coating of the present example was used instead of the coating mixture of Example

1. It was found that the weld spatter could be removed by wiping with a cloth.

Example 8.

Preparation (8) was prepared according to 5 the previously described method. Two rectangular blank sheets about 9 inches x 14 inches of an alloy of titanium with 2 / copper plus impurities were used in this example.

The blanks were treated as follows:

10 1. The metal surfaces, being particularly greasy were paraffin washed and degreased using an alkaline degreaser sold commercially under the trade name "Orthosil" (available from the Stavely Iron and Chemical Co. Ltd.) 15 followed by swilling in hot water and allowed to dry.

2. One of the plates was painted on one side with three equally spaced one inch stripes of the mixture prepared. The coating was 20 allowed to dry in air for 10 minutes. The

other blank was left uncoated.

3. The uncoated sheet was positioned about half an inch above the coated sheet, with the flat faces facing and the top sheet displaced 25 about one inch on the width. The coated force faced inwards.

4. The assembly was loaded into an electron heam welding machine (type 956 machine manufactured by Hawker Siddeley Dynamics 30 Ltd.) and the chamber was evacuated to a vacuum better than 1 \(\frac{1}{2}\) 10 7mm of Mercury. The assembly was located so that the electron beam would run parallel along the long face of the top sheet, and so that the line of the weld would miss the long edge of the bottom (costed) sheet by about 0.1 inches.

5. An electron heart was run along the long face of the top sheet, at a setting such that the sheet was fully penetrated and spatter was produced from the underside of the melt

run.

6. The conted sheet was then snaked in a complex alkaline cleaning agent (Ardrox 185 available from Ardrox Ltd) at 90-95° C for 45 30 minutes and was washed in hot water.

7. On examination after drying, it was seen that all the coating had been removed and that in the areas where the coating had been, no spatter had adhered to the metal. On the areas on the same side where there was no coating, spatter still adhered to the metal and could only be removed by mechanical means.

Example 9.

Preparation (9) was prepared according to 55 the previously described method. In this example, two sheet blanks of a martenesitic steel known as "Jethete" of a similar size to those i.; Example 8. The blanks were treated as follows:

1. The metal surfaces, being particularly greazy were paraffin washed and then degreased with trichloroethylene vapour to remove organic materials. Paint was removed by a suit-

able paint stripper.

2. As soon as possible after strip (1) one surface of one of the blanks was masked to leave three areas with the width of the blank. unsprayed. Three coats of the mixture were sprayed on to the remainder of the surface each coat being allowed to dry in air before the next was paplied. The other blank was left uncoated.

3. From this point onwards, the procedure was substantially similar to Example 8, from and including operation 3. The result was also

as in Example 8.

Example 10.

Preparation (10) was prepared according to the previously described method. In this example, two blanks of the nickel based alloy known as Nimonic 75 were used, and the preparation technique was substantially similar to that in Example 1.

The blanks were sprayed with "Stellite X40" a powder comprising a cobalt based alloy, which alloy also contains chromium, nickel and tungsten, (obtainable from Deloro Stellite Limited) from a plasma spray gun (3MB equipment, obtainable from Deloro Stellite Limited).

All other conditions and the final result were

as in Example 1.

Example 11.

Preparation (11) was prepared according to the previously described inethod. In this example, two blanks of an austenitic steel containing 18 // chromium and 8 // nickel were used and the preparation technique was substantially similar to that in Example 8.

The procedure and the final result were 100 substantially similar to those of Example 8

Although the invention has been described with reference to the foregoing examples in which the couting composition is painted or sprayed on to the surface to be protected, it 105 will be readily apparent to those skilled in the art and that other methods of application of the composition would be equally effective. Thus for example, it may be advantageous in some circumstances to apply the coating composition by dipping.

Whilst fusion welding, flame spraying and layer cutting have been exemplified as processes in which spatter is produced, compositions according to the present invention may 115 be used to protect any surface liable to be spattered with molten or semi-molten metal

and/or refractory particles.

#### WHAT WE CLAIM IS:—

1. A method of protecting at least part of 120 at least one surface which is positioned or is intended to be placed the vicinity of a metal and/or

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refractory spatter producing process from contamination by the spattered molten or semimolten products of such a process comprises the steps of coating said at least part of at least one surface with at least one layer of a composition comprising a mixture of an organophilic cation-modified (as hereinbefore defined) and a volatile organic vehicle, removing substantially all of said vehicle from said coating to leave a film of said clay on said at least one surface, effecting said spatter producing process, and removing any said spatter together with said film.

2. A method of protecting a surface as 15 claimed in claim 1 wherein said coating composition also contains an additive which is a non-volatile organic substance having adhesive properties and capable of forming a dry film when mixed with said organophilic cation-

20 modified clay.

3. A method of protecting a surface as claimed in claim 2 wherein said non-volatile organic substance is an acrylic resin.

4. A method of protecting a surface as claimed in claim 2 wherein said non-volatile organic substance is a silicone resin.

5. A method of protecting a surface as claimed in claim 2 wherein said non-volatile organic substance is a solid carboxylic acid.

30 6. A method of protecting a surface claimed in any preceding claim wherein said coating composition also contains an additive which is at least one non-volatile simple or complex refractory oxide having filling properties and capable of forming a dry film when mixed with said organophilic cation-modified clay.

7. A method of protecting a surface as claimed in any preceding claim wherein said organic vehicle has a boiling point of below

40 150° C at atmospheric pressure.

8. A method of protecting a surface as claimed in claim 7 wherein said boiling point is below 100° C at aumospheric pres-

9. A method of protecting a surface as claimed in any preceding claim wherein said

coating composition contains from 2 to 50 parts by weight of said organophilic cation modified clay, 50 to 300 parts by weight of said volatile vehicle, from 0 to 50 parts by weight of said non-volatile organic additives, and from 0 to 50 parts by weight of said non-volatile refractory oxide additive.

10. A method of protecting a surface as claimed ha claim 9 wherein the ratio of the weight of said organophilic cation modified clay to the weight of said non-volatile organic

additive is from 1:1 to 1.4.

11. A method of protecting a surface as claimed in any preceding claim wherein said film on said at least one surface is not less than 5 microns in thickness.

12. A method of protecting a surface as claimed in claim 11 wherein said film on said at least one surface is from 15 to 40 microns

in thickness.

13 A method of protecting a surface as claimed in any preceding claim wherein the organophilic cation-modified clay is dimethyl-dioctadecyl aminonium montmorillonite, do-decylamine montmorillonite or dimethyldioctadecyl ammonium hectorite.

14. A method of protecting a surface as claimed in any preceding claim wherein said spatter producing process is fusion welding.

15. A method of protecting a surface as claimed in any of claims 1—13 wherein said spatter producing process is flame spraying.

16. A method of protecting a surface as claimed in any of claims 1—13 wherein said spatter producing process is laser beam cutting.

17. A method of protecting a surface substantially as hereinhefore described and with reference to any of Examples 1—14 and the accompanying drawing.

18. Surfaces when protected by the method

of any preceding claim.

J. C. PURCELL, Chartered Patent Agent and Agent for the Applicants.

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COMPLETE SPECIFICATION

1 SHEET

This drawing is a reproduction of the Original on a reduced scale

